

**Gamma (K5 Based) Compressor Blade Material Design  
- Alpha Extrusion on a Small Scale**

Contract Period (22 April 2002 – 31 July 2003)

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**Final Report** (22 April 2002- 31 July 2003)

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## Statement of Work

**Purpose:** The objective of the 2002 task is to alpha-extrude ingots of two high temperature gamma alloys that were developed from the 2001 contract (C-70696-N). The alloys selected for study are 02K5-B1 (Ti-46Al-1Cr-3Nb-0.4W-0.15B-0.6C-0.2Si) and 02D-B1 (Ti-45.5Al-1Mn-6Nb-0.8W-0.15B-0.7C-0.2Si). The extrusions will be characterized and aged properly in order to generate improved balance in mechanical properties useful for advanced compressor blades. The aim is to develop a high temperature TiAl alloy for use in the Turbine Based Combined Cycle compressor. An aging study with 3 temperatures and 4 time periods at each temperature will be conducted to determine the optimum aging condition. High-magnification microscopic analyses shall be employed to define the size distribution of carbide as a function of aging temperature/time. The contractor shall define optimum materials' conditions by conducting tensile and creep testing for two over-aging conditions for both alloys and fatigue testing for one over-aging condition for each alloy.

## Tasks

**Starting Material Preparation:** Three ISM ingots shall be produced, two of 02K5-B1 and one of the 02D-B1 compositions. The ISM ingots shall be HIP'ed, homogenized, machined, and canned resulting in six 02K5 and three 02D billets of 2.35" x 5.5". An extrusion trial shall be conducted for each alloy, followed by microstructural evaluation, to determine the correct extrusion conditions. Combining wet-chemistry and DTA shall determine the composition. Five 02K5-B1 extrusions shall be produced under the modified conditions. Four 02K5-B1 extrusions will be round of 0.65"D by 55" useable length; the extrusion ratio shall be approximately 13:1. One 02K5-B1 extrusion shall be rectangular, with 0.7" x 0.7" x 32" dimensions and an extrusion ratio of approximately 8:1. Two 02D-B1 round extrusions shall be produced under the modified conditions with dimensions of 0.65" D x 55".

**Generation of Optimum Microstructures:** An aging study shall be conducted at 760°, 815°, and 900° C up to 192 hours. The microhardness of each sample shall be measured, and selected microstructural characterization by backscattered electron imaging and transmission electron microscopy shall be conducted. Three round extrusions (two 02K5-B1 and one 02D-B1) shall be given an aging treatment under the temperature/time combination specific to each alloy that was determined by the aging study.

**Mechanical Behavior Evaluation:** Properties shall be evaluated by tensile, creep, and fatigue testing. Each alloy will be over-aged with two selected conditions and tensile and creep testing will be performed. Tensile tests will be run at RT, 700°, and 870° C in air. Creep testing will be performed at 704° and 760° C. Duplicate tensile and creep samples shall be run for each test condition. High cycle fatigue will be run at an R-ratio of 0.1 at RT in air.

**Delivery:** Based on the microstructural and mechanical property evaluation, an aging condition that results in the best combination of properties shall be selected for each alloy. The remaining 2 round and 1 rectangular extrusions of 02K5-B1 and 1 round extrusion of 02D-B1 shall be aged according to the selected aging condition. The aged extrusion shall be delivered to NASA-GRC for evaluation. All unused material from the extrusions used for the microstructural and mechanical property evaluation shall also be delivered to NASA. The contractor shall provide a progress report every three months. A final report shall be delivered to NASA-GRC within 30 days after completion of technical effort.

## **1.0. Alloy Selection and Ingot Preparation**

Two gamma TiAl alloys were selected for this task and their nominal compositions are:

02K5-B: Ti-46Al-1Cr-3Nb-0.3W-0.2B-0.5C-0.2Si (Nominal)

02D-B: Ti-45Al-1Mn-6Nb-0.6W-0.2B-0.6C-0.2Si (Nominal)

One ingot for each alloy with 2.7" diameter was produced by the induction skull melting (ISM) technique at FlowServe, Dayton, Ohio. As will be shown later, these ingots contained coarse carbon- rich inclusions, and FlowServe kindly produced another set of ingots with an effort to remove the inclusions. The first set ingots are designated as 02K5-B1 and 02D-B1 and the second ones are designated as 02K5-B2 and 02D-B3. The compositions of these ingots analyzed and reported by at FlowServe are listed below. Clearly, the second set ingots have the carbon (C) contents are lower than those in the first ingots being close to the nominal.

**1st Set:**      02K5-B1: Ti-46.3Al-1.1Cr-3.0Nb-0.37W-0.22B-0.63C-0.17Si-0.25O (Actual)  
                  02D-B1: Ti-44.3Al-1.0Mn-5.8Nb-0.50W-0.18B-0.79C-0.20Si-0.24O (Actual)

**2nd Set:** 02K5B2: Ti-45.6Al-1.0Cr-3.1Nb-0.30W-0.18B-0.56C-0.20Si-0.22O  
                  02DB3: Ti-44.8Al-0.98Mn-6.0Nb-0.61W-0.22B-0.62C-0.19Si-0.21O

All ingots were given a hot isostatic pressing (HIP) at 1280°C for 6h under 207 Argon pressure. X-ray radiography conducted at FlowServe showed no pores present that are greater than roughly 0.5 mm in diameter.

## **2.0 Alpha Extrusion of the First Set Ingots**

Six (6) 02K5-B1 and two 02D-B1 billets having the dimensions of 2.35" (D) x 5.5" (L) were machined from the ingots. Eight cans, with one end open, to house these billets were also machined from solid stainless (ss) billet rods. Fig. 01a shows the billets, and cans along with ss lids. Each of these billets was inserted in a can, after wrapping with ceramic insulation cloth, and then a lid with evacuation tubing was welded on to the open end. The can was evacuated using a diffusion pump and then the tubing was closed mechanically and



cut short, as shown in Figure 1b. All evacuated and sealed billet cans were coated with boro-silicate by brushing.

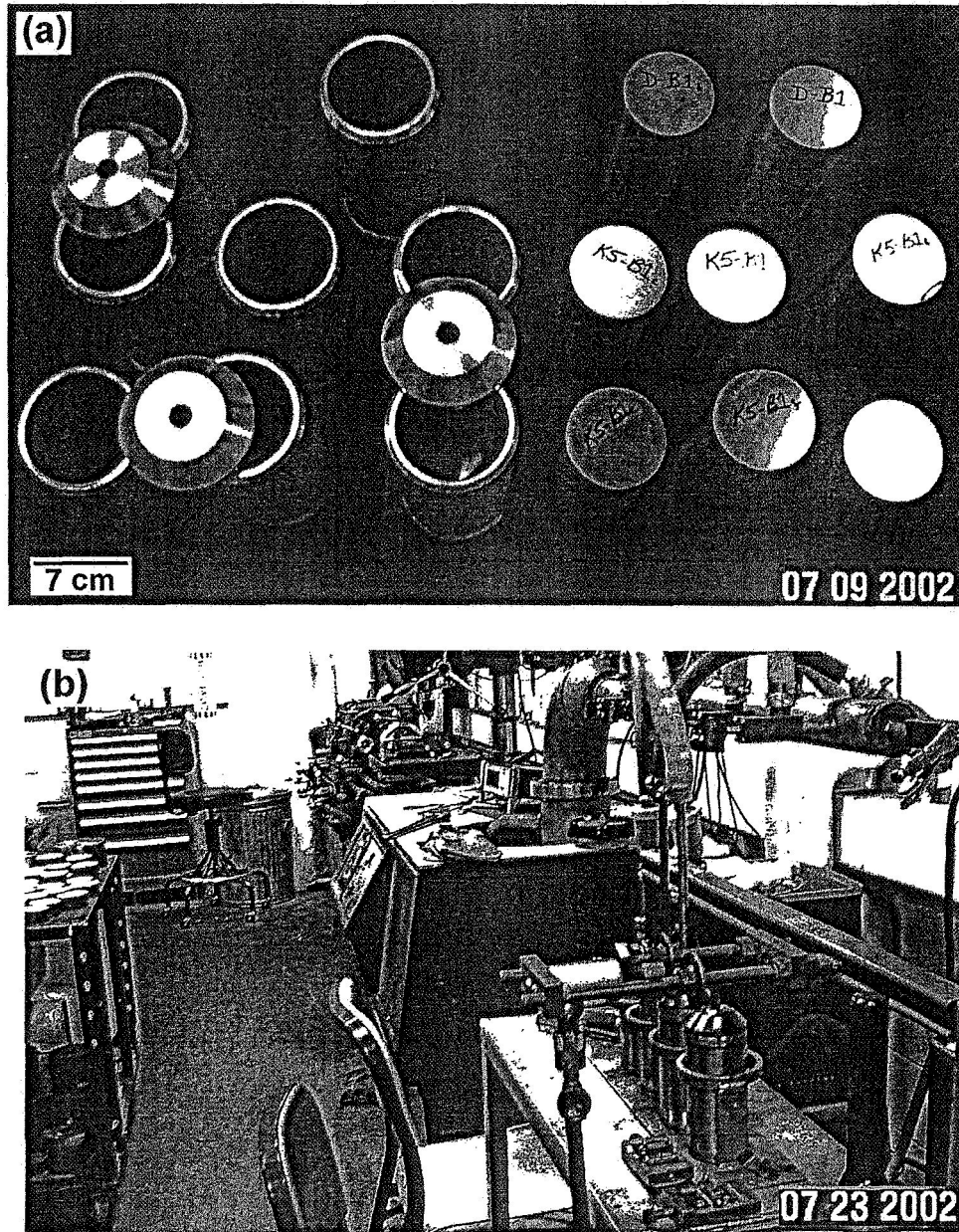


Figure 01: (a) Six 02K5-B1 and two (2) 02D-B1 billets and stainless steel cans, with one end closed, with three s.s. lids that will be welded onto the open ends. (b) Process of evacuation of sealed cans containing billets wrapped with ceramic insulation cloth. One evacuated billet can is shown to have the evacuation tubing closed and cut short.

Two 02D-B1 canned billets were alpha annealed at 1360°C for 1.6h in an air furnace and transferred to a ceramic brick on a steel table. Each canned billet was sat there for a total of 40 seconds (from the furnace), and then inserted in the extrusion die for extrusion. The time

elapse for the insertion till the onset of pushing was roughly 8 seconds. Fig. 02 shows pictorially the temperature decreases of the canned billets during the dwell. The alpha transus temperature ( $T_{\alpha}$ ) of alloy 02D-B1 with the nominal composition was estimated to be around 1300°C, indicating that the billet temperature is in the alpha phase field. Extrusion was conducted, using a computerized, hydraulic press having a 700 ton capacity, through a round exit hole die with a extrusion ratio of 18:1 at a pushing rate of roughly 5cm/sec. The extrusion process was successful with these conditions/parameters with relatively straight rods having c minute and then embedded into a container filled with silicel, mineral insulation powders. Five 02K5-B1 canned billets were extruded after soaking at 1380°C which is about 55°C higher than the estimated alpha transus of 1325°C. The first one was given the same dwell as that for 02D-B1 billets, and successfully extruded. However, the second one was stuck in the die during extrusion for unknown reasons, and because of this, a decision was made such that the remaining three 02K5-B1 billets would be given a shorter dwell, 30 seconds instead of 40 seconds. With this change, all three billets were extruded successfully, as shown in Fig. 03 where two technicians hold one of the three 02K-B1 billet extruded rods right after extrusion.

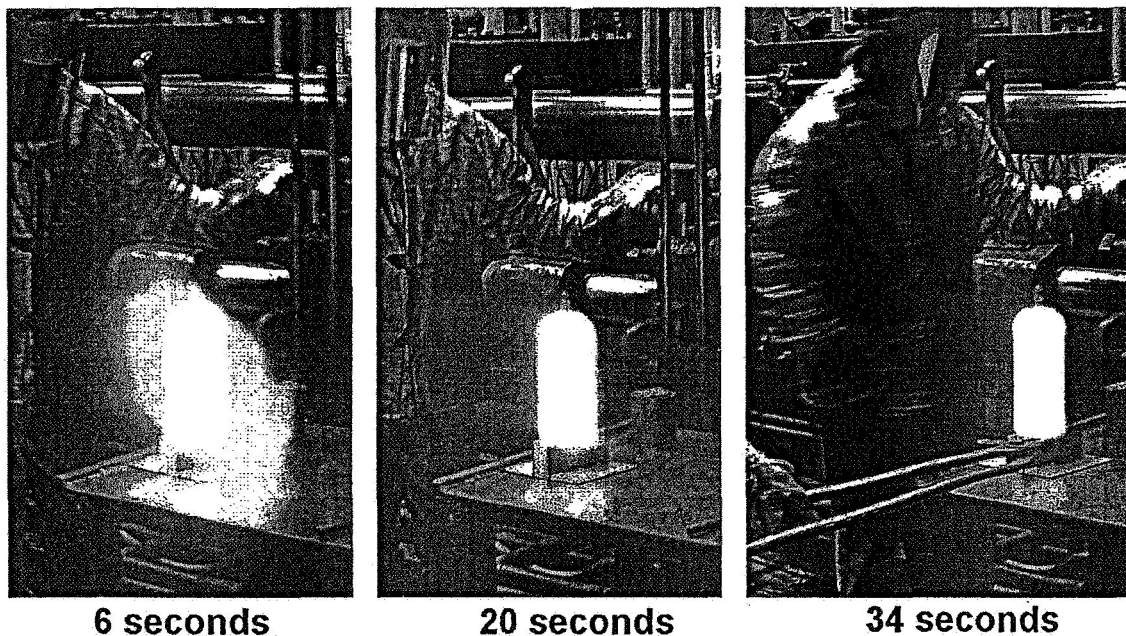


Figure 02: Cooling of 02D-B1 canned billets with time after being taken out of a furnace at 1360°C.

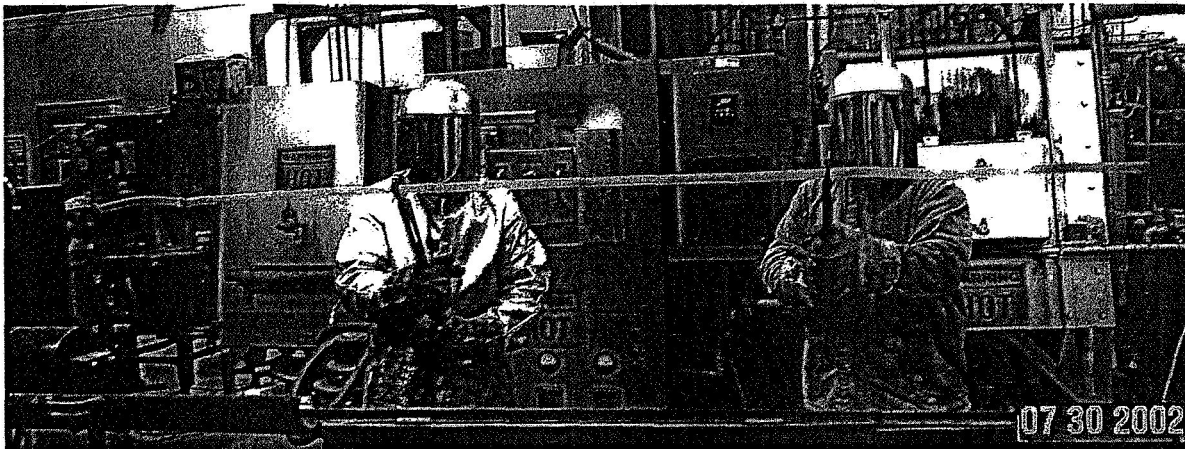


Figure 03: Right after extrusion, two technicians are holding the third 02K5-B1 extrusion. The billet was annealed at 1380°C and given 30 sec dwell and 8 sec inserting elapse. The yellowish glaring color indicates that the rod temperature may be still higher than 1200°C.

The extrusion conditions described above are summarized below.

Alloy Name	Soaking Temp. (°C)/Time (h)	Dwell (sec)	Extrusion Ratio (ER)
02K5B1	1380°C/1h	30	18:1
02DB1	1360°C/1h	40	18:1

Except one (02K5B1-b), all trials turned out to be reasonably successful to produce the TiAl rod diameter of 14mm, and the results are listed in Table 1. Three best looking extrusions, 0K5B1-a 02K5B1-c, and 02DB1-a, were conditioned by removing both ends, Figure 04, and delivered to GRC on 22 August 2002.

Table 1: Extrusion Results

Alloy	Ext. ID	Dwell (sec)	Useful Length (m)	Diameter ** (mm)	Remarks
02D-B1	02DB1-a	40	~1.5	14.0	To GRC
	02DB1-b	40	~1.5	14.0	
02K5-B1	02K5B1-a	40	~1.6	14.0	To GRC
	02K5B1-b	40		14.0	Failed
	02K5B1-c	30	~1.6	14.0	To GRC
	02K5B1-d	30	~1.6	14.0	
	02K5B1-e	30	~1.5	14.0	
	02K5B1-f	30	~1.5	14.0	

\*\* The TiAl material diameter was calculated based on the initial billet diameter (2.356" = 60.0mm) and the extrusion ratio (18:1). The actual diameter is expected to vary throughout the length, and it may not be perfectly round. However, machining specimens having a thread diameter of 0.5" (12.7 mm) should not be a problem.

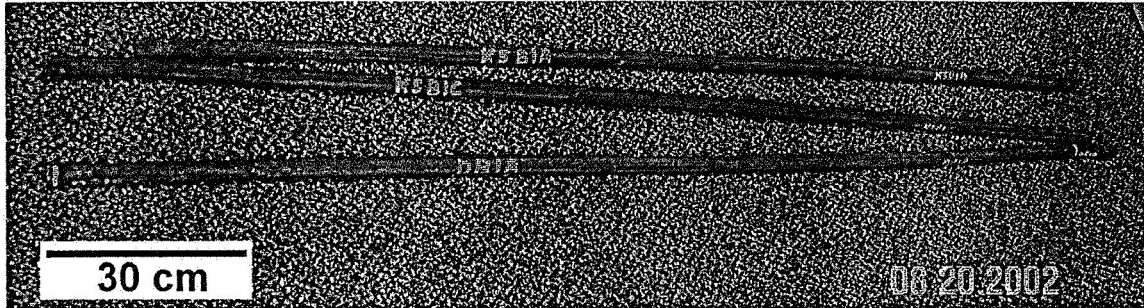


Figure 04: Two (2) 02K5-B1 and one (1) 02D-B1 extruded rods that were delivered to GRC in as extruded condition (08/22/02)

## 2.1. Alpha- Extruded Microstructures

Figure 05 and 06 show the typical as-extruded microstructures of 02KDB1 and 02K5B1, respectively, both being banded with slightly elongated to the extrusion direction, fine fully-lamellar grains. Both grain size (GS) ranges, as observed on the longitudinal section, are below  $100\ \mu\text{m}$  indicating that the alpha extrusion yielded refined lamellar grains that cannot be achieved by the conventional thermo-mechanical treatments. However, the 02K5B1 FL microstructure is coarser and slightly more elongated, with an average GS around  $70\ \mu\text{m}$ , than that (around  $40\ \mu\text{m}$ ) of 02DB1. As is clear from both figures, the grains are aligned in the extrusion directions, qualitatively indicating that a deformation texture may develop through dynamic recrystallization taking place during the extrusion process. Another feature observed in 02DB1 extrusions (Fig. 5) is the presence of bright needles (actually plates), as long as  $20\ \mu\text{m}$  or greater, that are generally aligned to the bands or extrusion direction. As shown later, these plates are tungsten and often contain boron. On the other hand, K5B1 alloy extrusions are shown to contain dark particles (Fig. 06) which are carbon-rich as will be shown later. It was found the former are intrinsic while the latter are inclusions that were formed due to an accidental use of large graphite particles at FlowServe, instead of fine ones, as a carbon source.



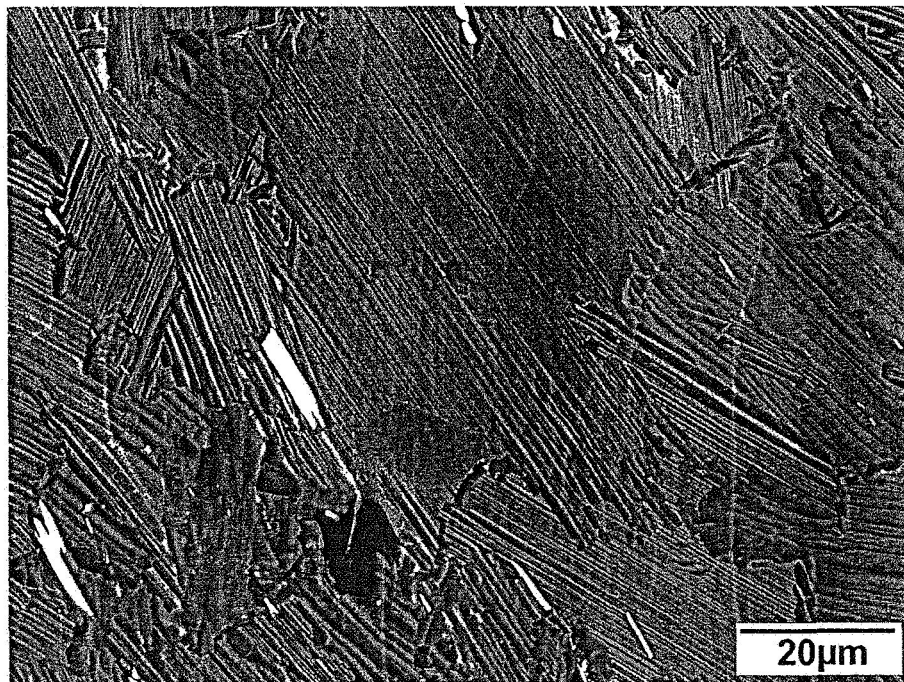
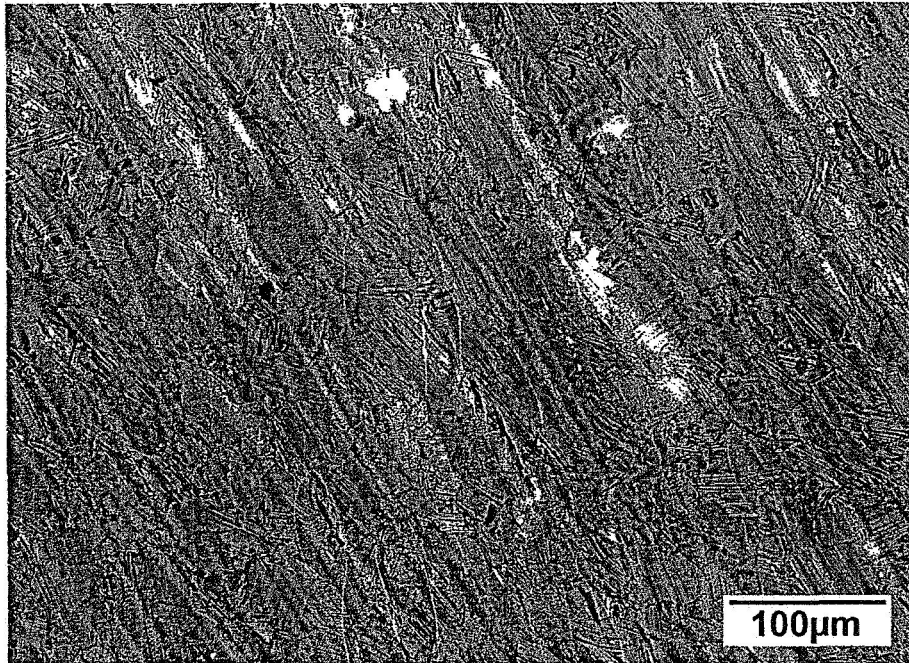


Figure 05: BEI microstructures of the longitudinal section of as-extruded 02DB1 alloy rod at two different magnifications, showing band structures of fine ( $<70\mu\text{m}$ ) lamellar grains dispersed with apparent inclusions (imaged dark) and their stringers aligned to the extrusion direction.

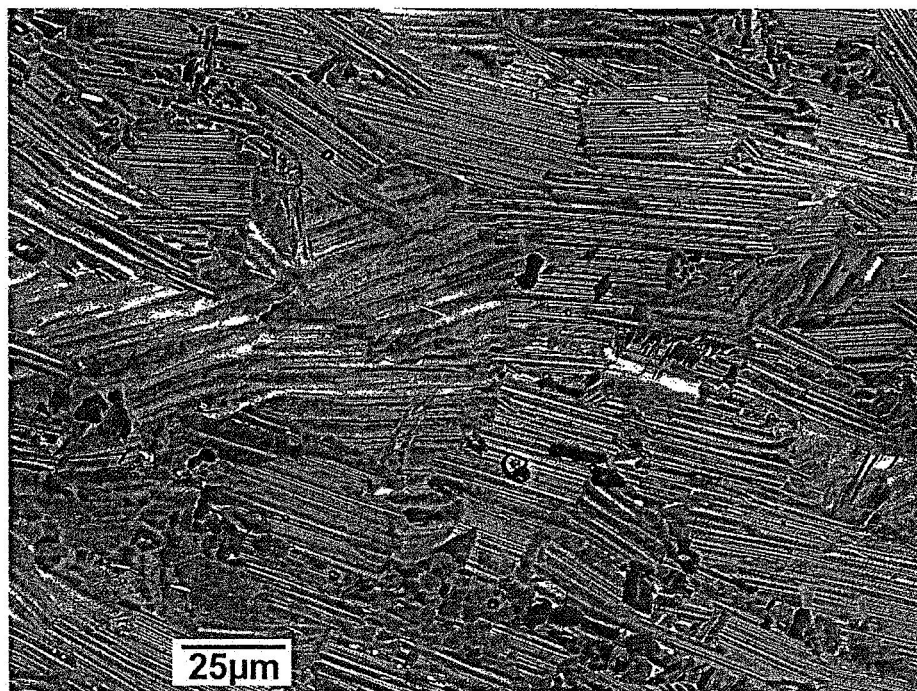
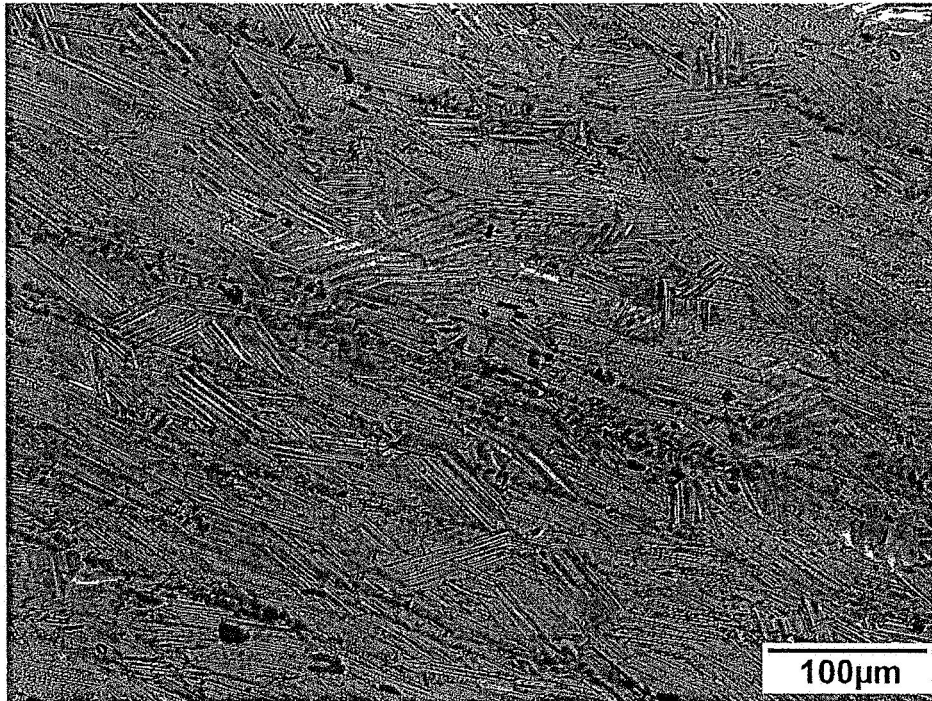


Figure 06: BEI microstructures of the longitudinal section of as-extruded 02K5-B1 alloy rod at two different magnifications, showing band structures of fine ( $<100\mu\text{m}$ ) lamellar grains dispersed with apparent inclusions (imaged dark) and their stringers aligned to the extrusion direction.

## 2.2 Aging Experiments of Selected Extrusions

One extrusion rod for each alloy was selected to characterize the as-extruded microstructure and microstructural variations upon aging. These extrusion rods are 02K5B1-f and 02DB1-

b. Aging treatments were selected as listed in Table 2.

Table 2. Aging Conditions for 02K5B1-f and 02DB1-b Extrusions

Alloy 02K5-B1f Extrusion	Alloy 02D-B1b Extrusion
02K5-B1f-00: No Aging	02D-B1b-00: No Aging
02K5-B1f-01: 900°C/24h/AC	02D-B1b-01: 900°C/24h/AC
02K5-B1f-02: 900°C/48h/AC	02D-B1b-02: 900°C/48h/AC
02K5-B1f-03: 900°C/96h/AC	02D-B1b-03: 900°C/96h/AC
02K5-B1f-04: 1000°C/4h/AC	02D-B1b-04: 1000°C/4h/AC
02K5-B1f-05: 1000°C/24h/AC	02D-B1b-05: 1000°C/24h/AC
02K5-B1f-06: 1000°C/48h/AC	02D-B1b-06: 1000°C/48h/AC

When observed at a low magnification (<200x) under polarized light (PL) conditions, the aging treatments at the conditions listed in Table 2 did not appear to alter the as-extruded microstructures appreciably for both alloy extrusions. However, the observations made using the back-scattered electron imaging (BEI) technique show that alpha-2 lamellar plates are broken up into particles as shown for an aging (900°C/48h) (Figure 07 for 02K5B1-f and Figure 08 for 02DB1-b). Upon this aging treatment, 02K5B1 extrusions are recrystallized extensively (Fig. 07a), while recrystallization of 02DB1 extrusions is relatively localized (Fig. 08b).

As is clear from Fig. 08b and previous analysis of X-ray mapping, aging at 900°C/48h/AC transforms alpha-2 plates into particles and rods of alpha-2 (light), B2 (bright), and Ti2AlC carbide (dark), Fig. 08b. The transformation is analyzed to be the result of the equilibration process (aging), which involves thinning (reduction in volume) of alpha-2 plates, saturation and segregation of carbon, breaking up, and generation of carbides. The net result is that the lamellar structure becomes coarse and may lose some of its original anisotropy nature (coming from the original near-perfect lath structure). The breaking process appears to be completed at 900°C/48h for alloy 02K5B1 extrusions, while it requires additional aging for a longer period or at higher temperatures for 02DB1 extrusions.

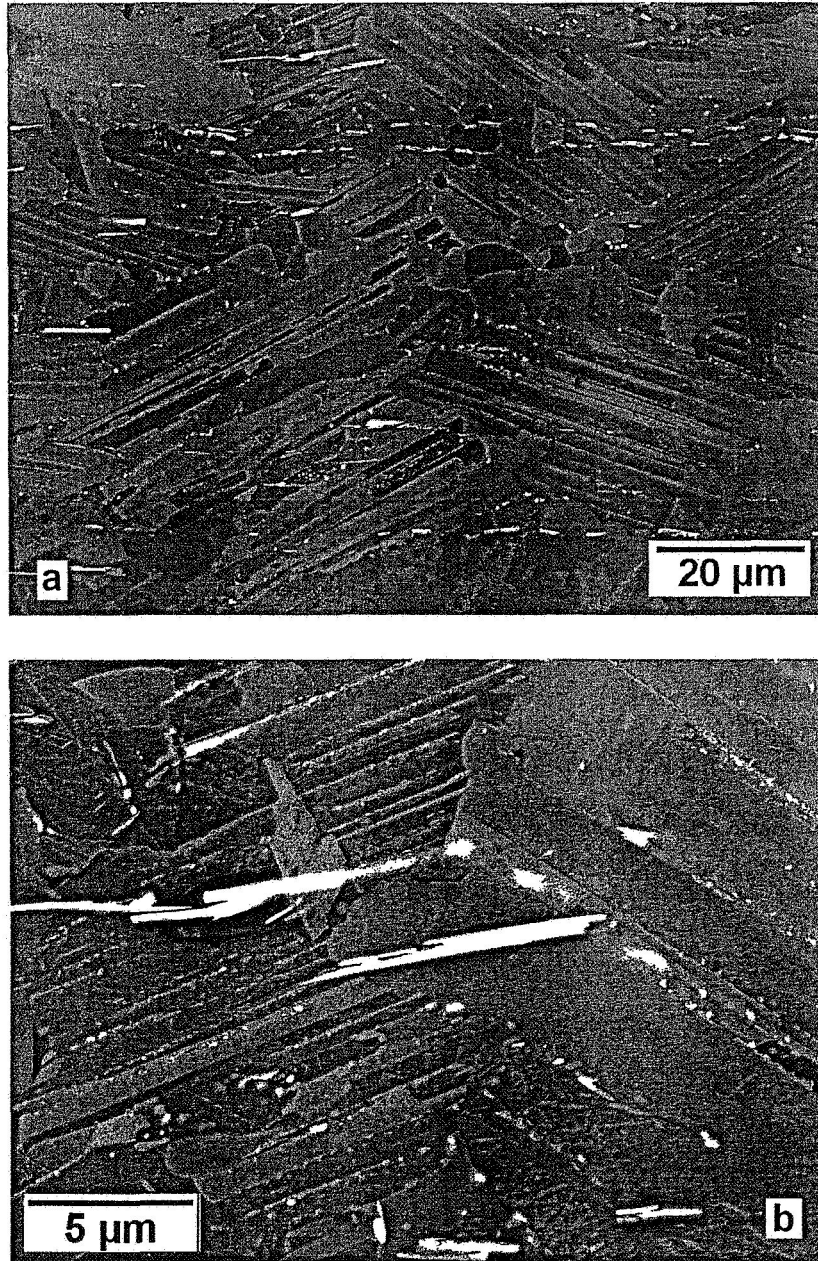


Figure 07: BEI microstructures of the longitudinal section of extruded and aged (900°C/48h) 02K5-B1 alloy rod at two different magnifications, showing recrystallization of the gamma phase matrix (a) and breakup of alpha-2 laths into particles (imaged bright) (b). Coarse needles/plates (imaged bright) are rich with tungsten and boron and aligned in the extrusion direction.



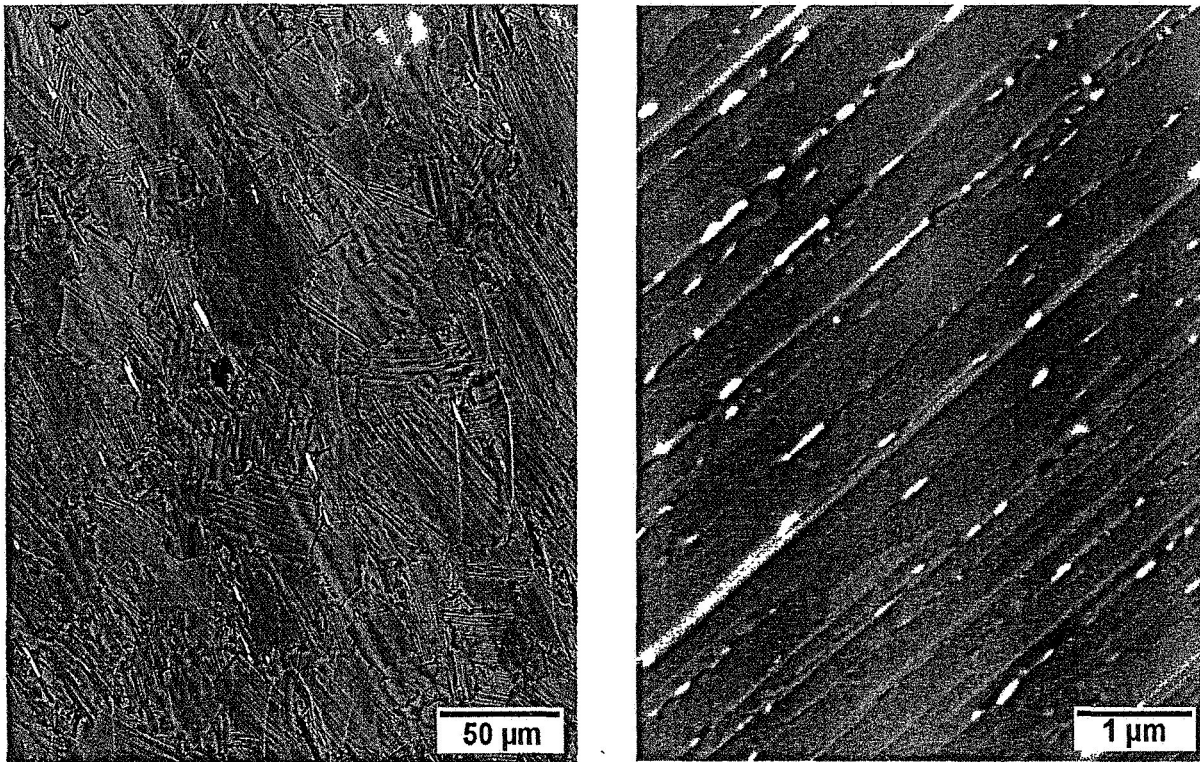


Figure 08: Back-scattered electron images of 02DB1 alpha extrusion aged at 900°C/48h at low magnification (a) and high magnification (b). The magnified view shows the alpha-2 laths are broken up, resulting in the formation of arrays of alpha-2 (bright), B2 (brighter), carbide (dark) and silicide (light) particles.

One important observation is that both alloy materials contained coarse carbon-rich rod-like particles, imaged gray or dark, sometimes in the form of arrays or stringers. EDS analysis and X-ray mapping experiments show that these are rich with carbon and titanium in 02K5B1 alloys, Figs. 09. These particles were then assumed to be a Ti-carbide. Fig. 10 shows the BEI of 02DB1b alpha extrusion microstructure after aging at 900°C/96h. The material is shown to contain inclusions such as large, blocky graphite particles (imaged dark) in (a), Ti-oxides imaged gray in (a) and (b), and W-rich needles (imaged bright). These inclusions were identified through EDS analyses and X-ray mapping.

The presence of these unwanted particles were serious enough to discuss with Scott Reed of FlowServe who produced the ISM ingots. Scott Reed admitted their mistake in that the foundry people used large graphite chips for melting the alloy ingots instead of fine, carbon particles. This incident happened, according to Scott, because at that time they could not find the fine carbon particles and the technicians were not aware of the implications.

FlowServe agreed to reproduce the ingots again free of charge. We did not know how detrimental effects the large Ti-C particles might be, but it was assumed that the major effect would be to reduce ductility and fatigue life. Nevertheless, a recommendation made to GRC was to conduct tensile testing of the provided materials, and if the results are reasonable, then creep testing. In the meantime, the second batch extrusions will be ready for delivery.

### 02K5B1f: Alpha Extruded + Aged (900°C/48h/AC)

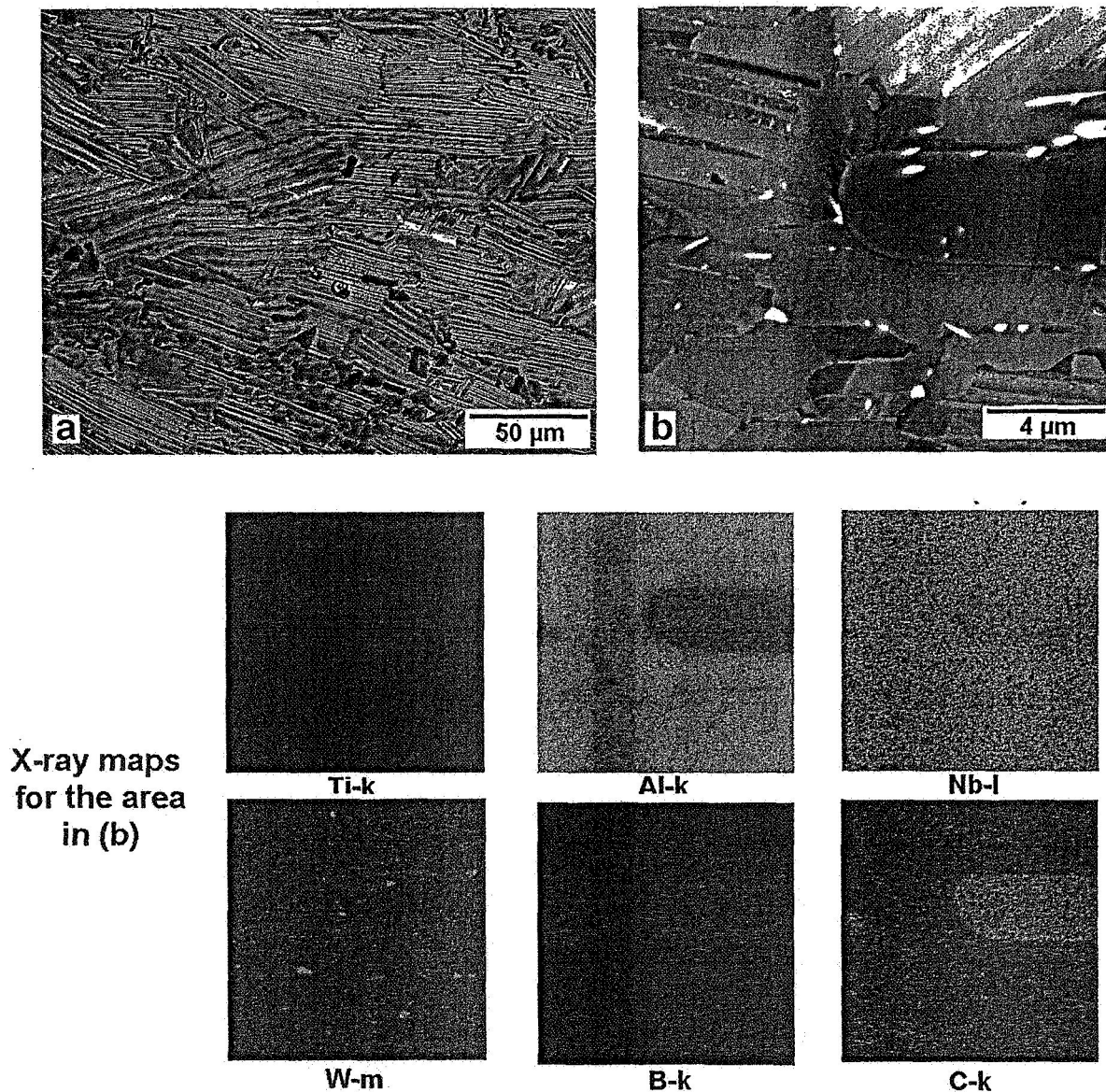


Fig. 09: 02K5 extrusion aged at 900°C/48h, showing the BEI of the overall extrusion microstructure (a), magnified BEI of the area containing dark, elongated particles (b), X-ray maps for this area (c). Clearly, the coarse, gray parcels in (b) are rich with C and Ti. These are generally rod-shaped and considered to be produced from large graphite chips that were wrongly added as the carbon source.

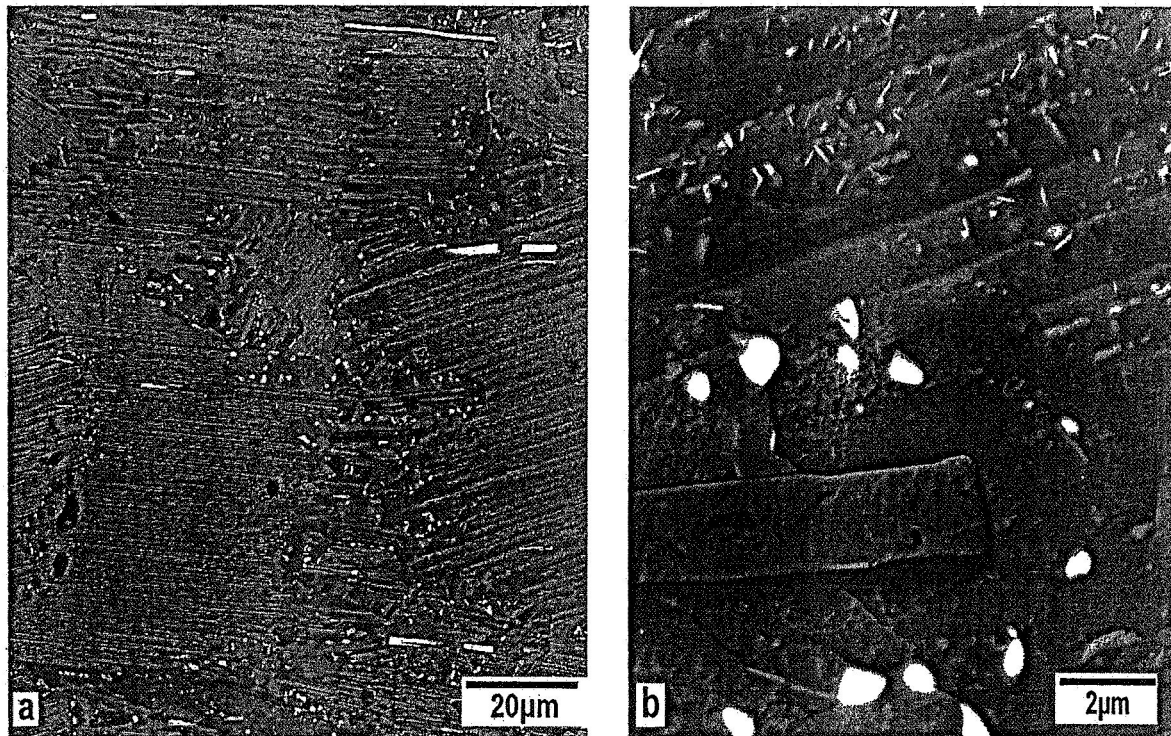


Figure 10: Back-scattered electron images of 02DB1b alpha extrusion aged at 900°C for 96h at low magnification (a) and high magnification (b). Two typical inclusions are graphite-rich (gray and blocky) and tungsten-rich (imaged bright).

### 2.3 Tensile Properties

Tensile properties were conducted on both extrusions in both A0 (as extruded) and A48 (aged at 900°C/48h) conditions at RT, 760°C, 870°C and 980°C in air. The results are listed in table 3, where the results from alloy 99C extrusion are also listed for comparison. Alloy 99C was produced in 1999 as an alloy K5 variation with the composition of Ti-46.5Al-2Cr-3Nb-0.2W-0.2B-0.2C, and was extruded at three different extrusion ratios. The 99C alloy extrusions were tested in the as-extruded condition only.

Table 3. Effect of Extrusion Ratio and Aging Condition on Tensile Properties

ID	Aging	ER	T (°C)	YS (MPa)	UTS (MPa)	Sf (%)	μ (GPa)
02K5B1f	A0	18	23	1044	1090	1.00	163
02K5B1f	A0	18	760	692	875	9.20	149
02K5B1f	A0	18	870	608	704	28.18	149

02K5B1f	A48	18	23	661	802	2.65	193
02K5B1f	A48	18	760	690	690	25.1	163
02K5B1f	A48	18	870	467	514	52.1	129
02DB1b	A0	18	23	---	940	0.59	168
02DB1b	A0	18	760	823	1045	1.40	172
02DB1b	A0	18	870	720	856	1.45	123
02DB1b	A0	18	980	525	611	9.12	147
02DB1b	A48	18	23	1055	1090	1.20	168
02DB1b	A48	18	760	683	878	7.61	157
99C1	A0	25	23	---	536	0.35	170
99C2	A0	16	23	841	869	0.92	176
99C3	A0	9	23	796	881	2.10	171

ER: Extrusion Ratio; A0: No Aging or As-extruded; A48 = Aged 900°C/48h/AC

Remarkable strength levels were achieved for 02K5B1 extrusion, with both yield strength (YS) and ultimate tensile strength (UTS) levels being above 1000MPa at RT and YS=608MPa and UTS=704MPa at 870°C. Aging at 900°C/48h reduced the strength levels (which, however, are still remarkably high), but increased the RT strain-to-failure from 1.0 to 2.65%! This RT ductility increase appears to be involved in the reduction of the BDTT. On the other hand, 02DB1 alloy extrusions are generally show poor RT ductility in as extruded condition. The specimens often fail elastically at RT, but aging at 900°C/48h the ductility increase the RT strain-to-failure, as does for 02K5B1, to 1.2%. Alloy 02DB1 extrusions show greater strength levels and greater high-temperature strength retention than 02K5B1 extrusions. The BDTT appears to be below 760°C for 02K5B1 extrusions whereas it ranges above 870°C for 02DB1 extrusions in as extruded conditions. Both BDTT's are reduced substantially upon aging. These observations are shown in Fig. 11. Ductility appears to depend on extrusion ratio as well, and the best ductility was achieved at ER~9:1 for alloy 99C extrusions. If this is the case, both K5B and DB1 extrusions are expected to exhibit greater ductility at lower extrusion ratios.

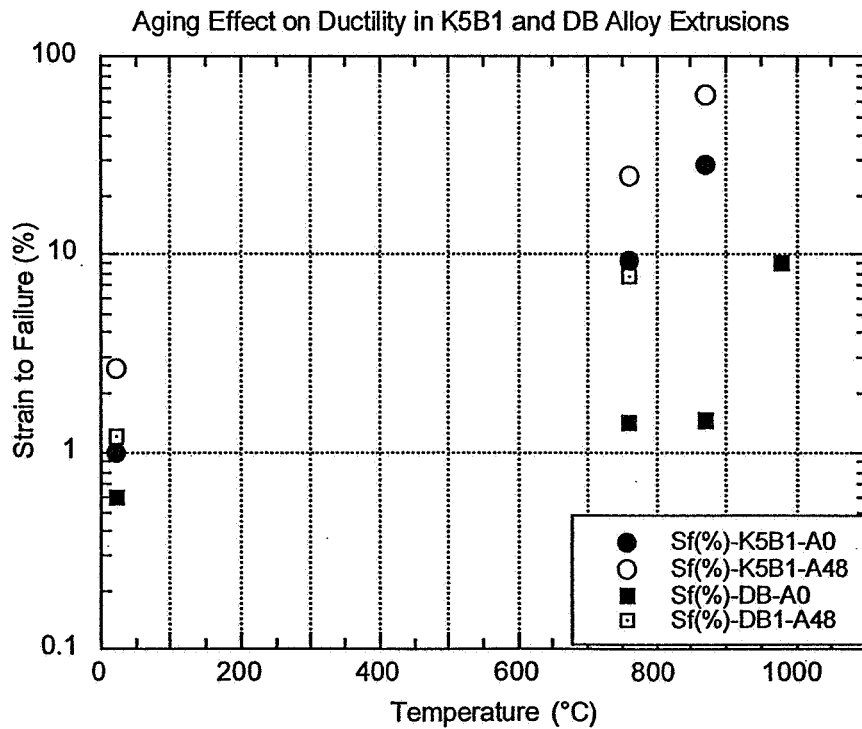
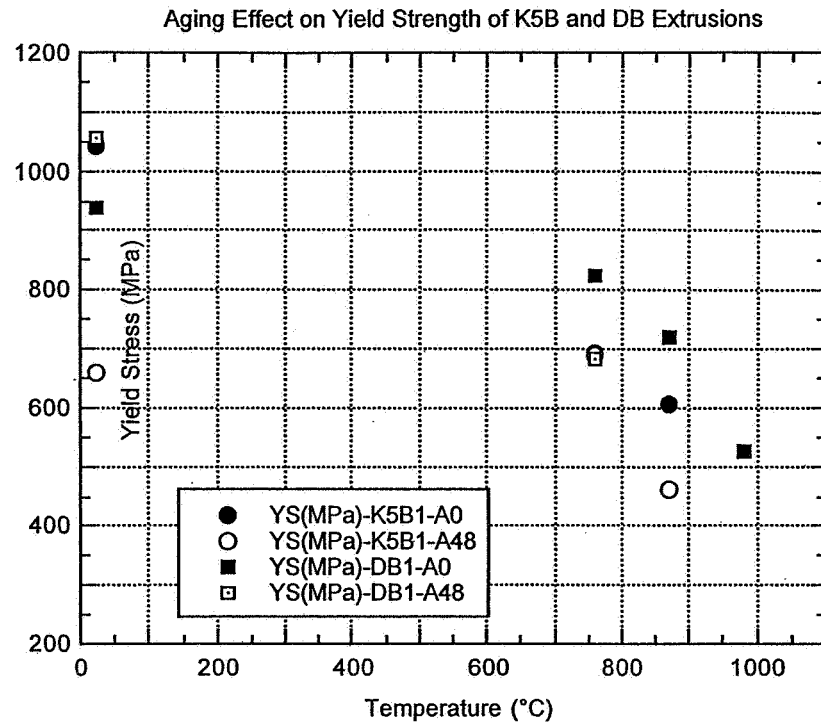


Figure 11: The effect of aging (900°C/48h) on yield strength and total elongation (strain to failure) is shown as a function of test temperature. DB1 extrusions exhibit higher strength and lower ductility throughout the test temperature range. The aging lowers strength levels and increases the BDDT's.



### 3.0 Alpha Extrusion of 2<sup>nd</sup> Set Ingots

Due to the large inclusions detected in the first set alloy ingots, as described earlier, FlowServe produced two more ingots with no cost. They are:

02K5-B2: Ti-45.6Al-1.0Cr-3.1Nb-0.30W-0.18B-0.56C-0.20Si-0.22O  
02D-B3: Ti-44.8Al-0.98Mn-6.0Nb-0.61W-0.22B-0.62C-0.19Si-0.21O

These alloys will be designated as KB2 and DB2, respectively, for convenience sake. Seven extrusions were produced using the alpha-extrusion technique, as follows:

Three (3) KB2 round-bar extrusions at 17:1 ER after soaking at 1390°C for 1.5hr

Two (2) DB3 round-bar extrusions at 17.5:1 ER after soaking 1370°C for 1.5hr

Two (2) KB2 square extrusions at 6.5:1 ER after soaking at 1390°C for 1.5hr

Dwell: 35 sec

Two round KB2 and one DB3 extrusions were delivered to GRC on 30 October 2002, and two rectangular KB2 extrusions on 15 November 2002

### 3.1 Microstructure Evolutions and Characterization

As was the case in 02KB1 extrusions, alpha extruded lamellar grains are finer than 100 µm and randomly oriented on transverse planes. However, their laths are clearly oriented to the extrusion direction, as observed in BEI conditions, Fig. 12. A stronger texturing appears to be developed in DB3 extrusions, Fig. 13, where severe mixing of material is shown to have taken place during extrusion. Inclusions such as carbon-rich or oxide particles were not observed; indicating use of large graphite chips was the cause of introducing such particles in the extrusions of the first group alloys (02K5B1 and 02DB1). Aging experiments were conducted on both alloy extrusions, as follows:

KB2-00: As Extruded	
KB2-01: 900°C/24h (L/T)	KB2-11: 1000°C/7h (L/T)
KB2-02: 900°C/48h (L/T)	KB2-12: 1000°C/24h (L/T)
KB2-03: 900°C/96h (L/T)	KB2-13: 1000°C/48h (L/T)
DB3-00: As Extruded	
DB3-01: 900°C/24h (L/T)	DB3-11: 1000°C/7h (L/T)
DB3-02: 900°C/48h (L/T)	DB3-12: 1000°C/24h (L/T)
DB3-03: 900°C/96h (L/T)	DB3-13: 1000°C/48h (L/T)

L: Longitudinal (Surface); T: Transverse (Surface)

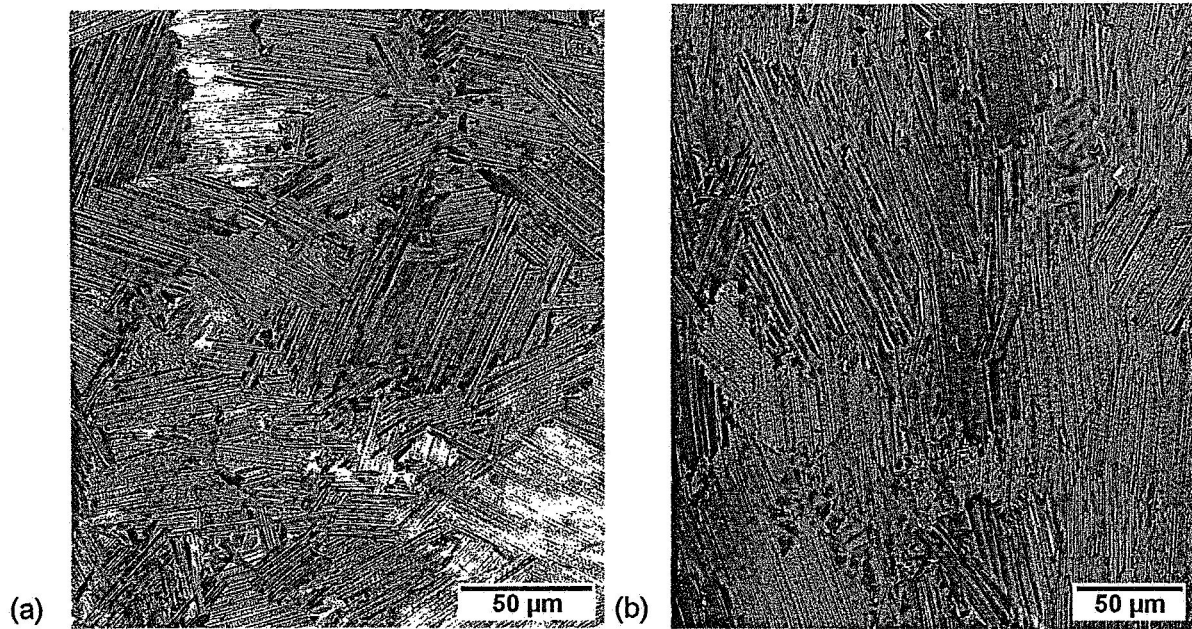


Fig. 12: BEI microstructures of KB2 extrusion in transverse (a) and longitudinal (b) directions showing random orientation of alpha lamellar grains on transverse planes and texturing of laths in the extrusion direction.

BEI of DB3 Alpha-Extrusion after Aging at 900°C for 24h

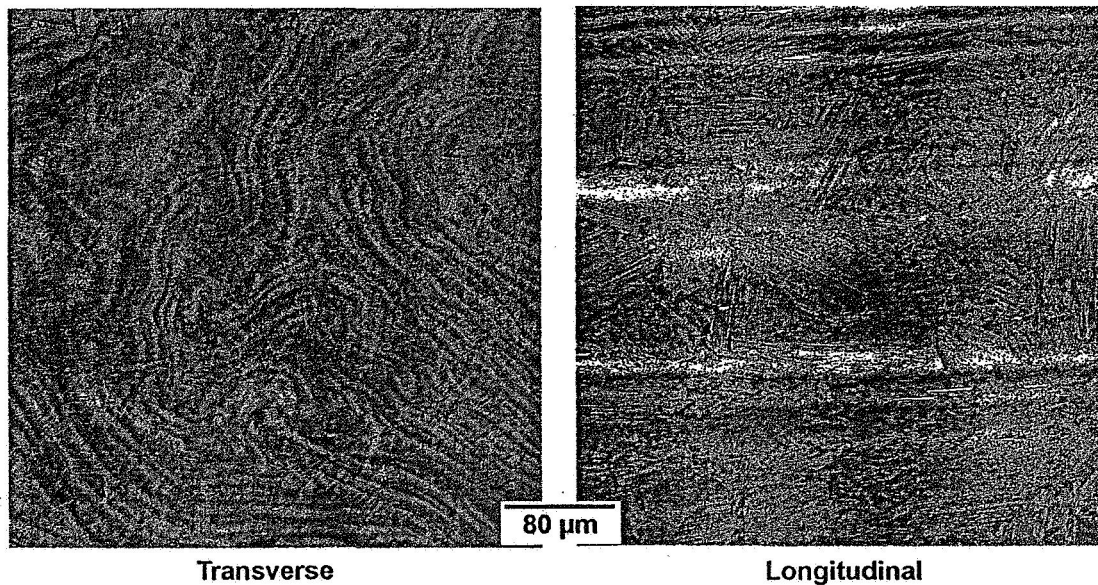


Fig. 13: BEI microstructures of DB3 extrusion in transverse and longitudinal directions showing mixed material flow on transverse planes and strong texturing of laths in the extrusion direction.

As expected, aging treatments destabilized alpha-2 laths and broke them into particles, as shown for KB2 extrusion aged at 900°C for 48h, Fig. 14. Breakup appears to be completed

in KB2 extrusions upon this aging. DB3 extrusion appears to respond to aging similarly, Fig. 15a, where an area shows nearly complete breakup of alpha-2 plates upon aging at 900°C for 48h. For both alloy extrusions, further aging results in recrystallized gamma grains and coarsening of lath space and alpha-2 particles, as shown for DB3 extrusion aged at 1000°C for 48h, Fig. 15b. In Fig. 15b, carbide particles (imaged dark and often elongated) are shown to be present to form arrays with alpha-2 particles (imaged bright).

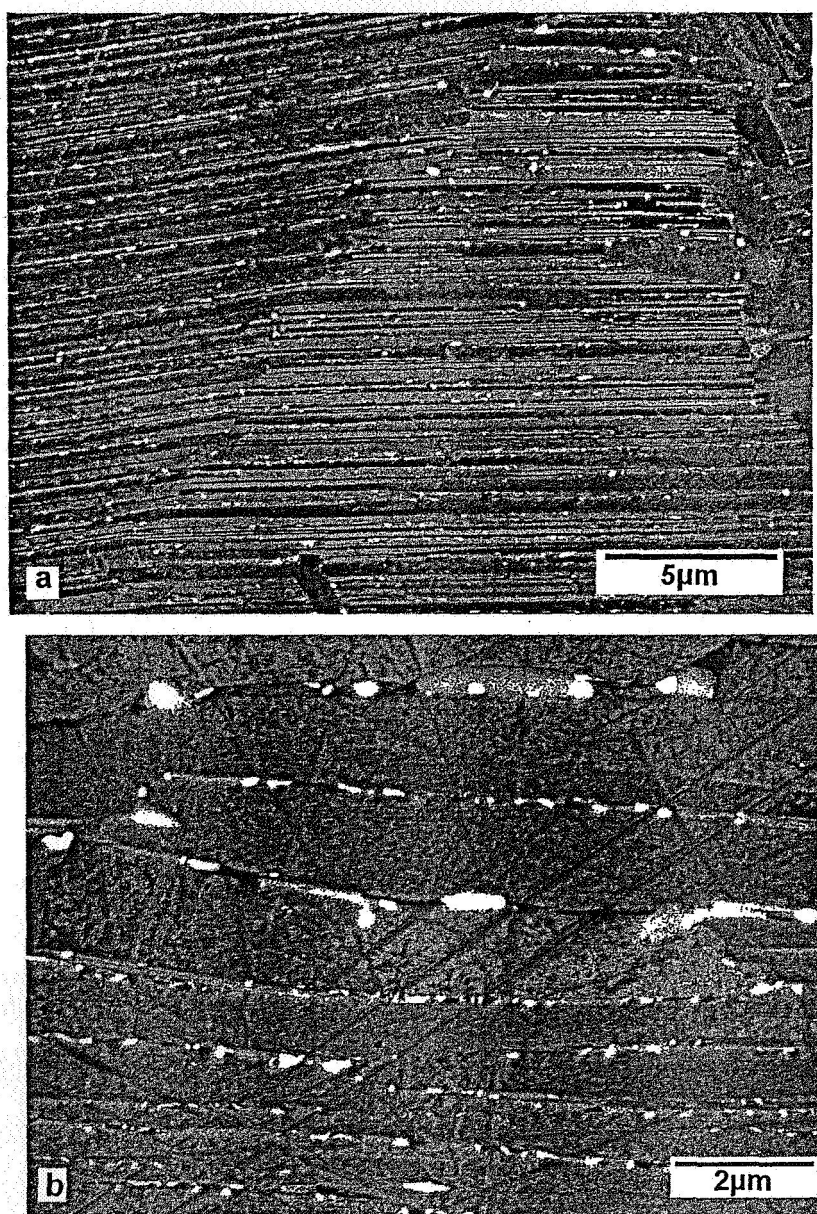


Figure 15: BEI images of DB3 extrusion after aging at 900°C for 48h (a) and 1000°C for 48h (b) showing complete breakup of alpha-2 laths (a) and coarsening of the breakups and lamellar spacing (b). Carbide particles, often elongated and imaged dark, are shown to form arrays with broken alpha-2 particles or plates (imaged bright).



### 3.2 Mechanical Property Evaluation

Mechanical properties were tested on both alloy extrusions in the following aging conditions:

KB2-A2: 900°C/48h/FC/500°C/AC  
 DB3-A1: 900°C/24h/FC/500°C/AC  
 DB3-A2: 900°C/48h/FC/500°C/AC

In addition, selected tensile specimens were given an exposure (800°C for 200h in air) and tested at RT with or without polishing the gage section. Hand polishing with 800 mesh sand paper was used in an effort to remove oxide scale that forms during the exposure. Recently, Susan Draper, GRC, has observed that when exposed at the same condition, Plansee ( $\gamma$ -met) extrusion specimens lose the RT ductility completely. She has observed this embrittlement phenomenon after 1000°C/2h exposure as well, and furthermore even when the treatment was conducted in vacuum. In these cases, the starting g-met materials were either in the as received (extruded NFL) condition or given an alpha treatment for FL. Understanding this behavior is critically important and has to be investigated. Test matrices are listed in Table 4.

Table 4: Mechanical Test Matrices

	<u>Tensile</u> (0.25"D-0.13"G)	<u>Creep</u> (0.375"D-0.25G)	<u>Fatigue</u> (0.375"G-Flat G)
	T (°C) (number)	T (°C) (n)	T (°C) (n)
KB2-A2	RT (1) RT (E) RT (EP)	760 (2)	
DB3-A1	6 RT (2) RT (E) RT (EP) 704 (1) 815 (1)	4 760 (1)	3 RT (2)
DB3-A2	6 RT (2) RT (E) RT (EP) 704 (1) 815 (1)	4 760 (2)	3 RT (3)

E: Exposed in air at 800°C for 200h

EP: E + Hand Polishing with 800 mesh sand paper

### 3.2.1 Tensile Properties

Tensile properties measured on both alloy specimens are listed in Table 5, along with their aging and post-aging treatments.

Table 5: Tensile Properties of KB2 and DB3 Extrusions as a Function of Test Temperature under Different Post-Extrusion Aging Conditions and Post-Exposure Treatments

Sample ID	Age / Exp	Test T (°C)	YS (MPa)	Yield Strain(%)	UTS (MPa)	Strain to Failure (%)
KB2-A2	A2	23	763	0.65	890	2.27
DB3-A1	A1	23	948	-	948	-
DB3-A1	A1	23	839	0.95	918	1.42
DB3-A2	A2	23	878	1.27	929	1.70
DB3-A2	A2	23	847	0.78	957	1.73
DB3-A1	A	704	686	0.68	868	3.40
DB3-A2	A2	704	665	0.61	868	2.33
DB3-A1	A1	815	630	0.59	737	19.0 +
DB3-A2	A2	815	660	0.75	754	14.10
KB2-A2E	A2 + Exp	23	721	0.62	755	1.40
DB3-A1E	A1 + Exp	23	-	-	576	0.35
DB3-A2E	A2 + Exp	23	-	-	566	0.38
KB2-A2EP	A2 + Exp + P	23	761	0.67	792	0.95
DB3-A1EP	A1 + Exp + P	23	-	-	599	0.41
DB3-A2EP	A2 + Exp + P	23	-	-	668	0.44

A1 and A2: Aging (see text); Exp: 800°C for 200h; P: Hand polishing

As shown, the aged KB2 specimens have lower strengths and greater ductility than DB3 extrusions at any temperatures tested. Clearly, the exposure reduced the RT ductility of aged DB3 specimens; however, KB2 specimens still maintain reasonable RT ductility. Hand

polishing of KB2 specimens apparently reduced RT ductility for unknown reasons. The response to the exposure and polishing is plotted for both alloy specimens in Fig. 6.

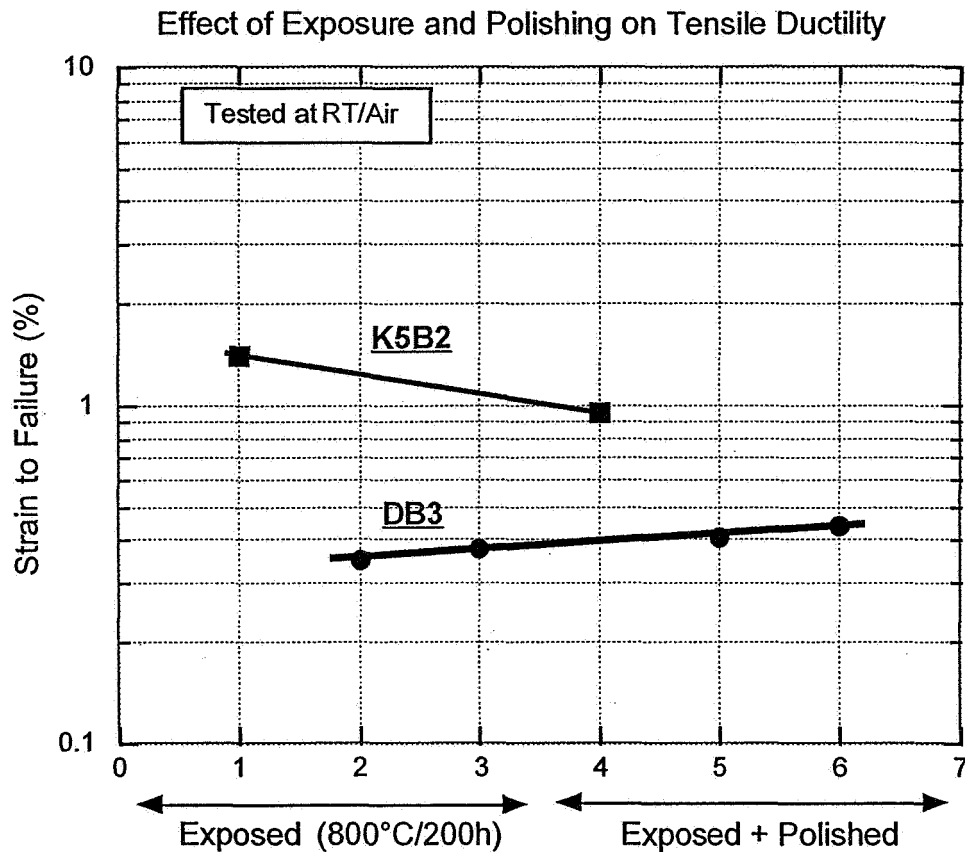


Figure 16: Exposure and subsequent polishing on RT ductility for aged KB2 and aged DB3 extrusion specimens. DB 3 specimens were failed with the elasticity limit, whereas KB2 specimens still retain reasonable ductility. Hand polishing on the gage section reduced the ductility of KB2 specimens.

### 3.2.2 Fatigue Properties

Fatigue testing was conducted at RT at  $R=0.1$  on 02DB3 smooth flat gage specimens in two different aging conditions. The results are plotted in Fig. 17, where the max stress at the first cycle was assumed to be the ultimate tensile strength of the material. Both aging conditions resulted in typical, conventional SN curves with gradual decrease in fatigue stress with cycle. Some scattering is observed, perhaps, due to the nonuniform microstructures that are hardly removable in the current ISM ingot melting technology for high-Nb containing alloys. It appears that lengthening aging time may reduce the overall fatigue stress. There

is some indication that there exists the fatigue strength for  $R=0.1$ , which is around 600 MPa at ten million cycles.

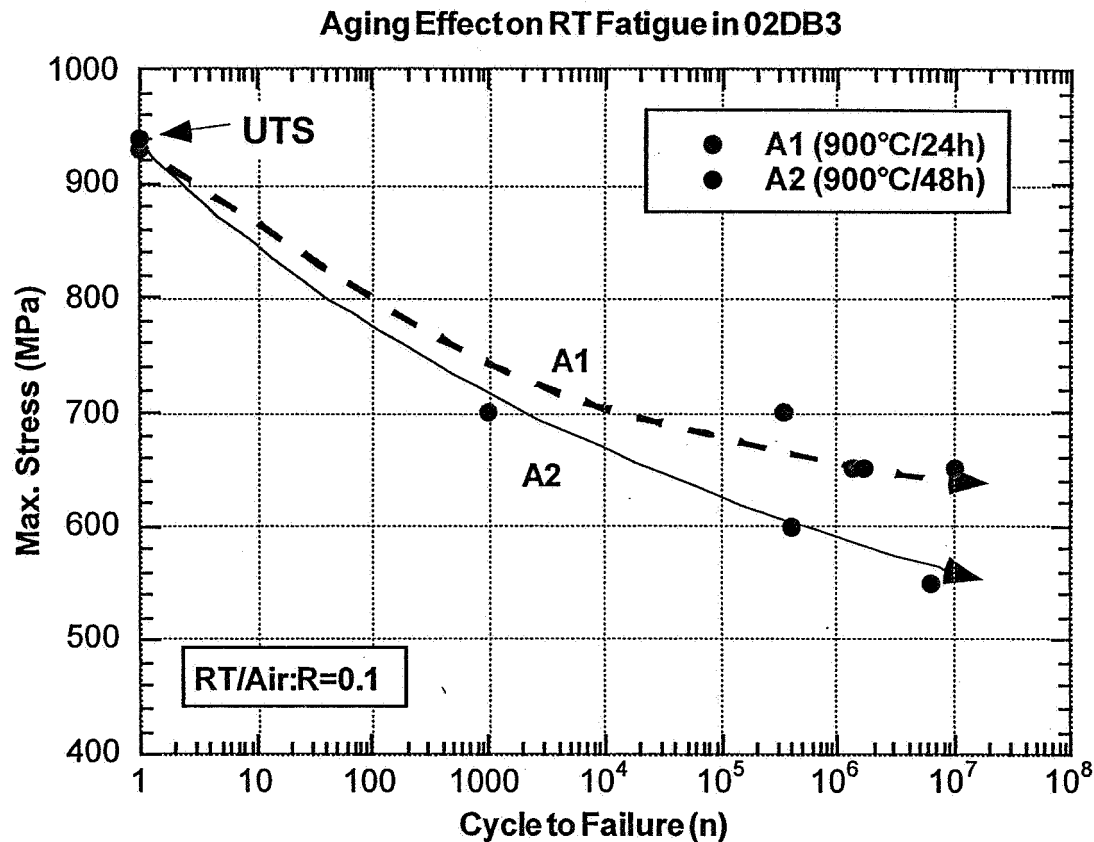


Figure 17: Fatigue properties of DB3 specimens as a function of cycle to failure under two aging conditions. Test was conducted at  $R=0.1$  and in air.

### 3.2.3 Creep Properties

Two types of creep tests were conducted: one under a constant, high stress (276MPa) and the other under step-by-step stress increase conditions. The results from the constant stress test are plotted in Fig. 18 and also listed in Table 6. It is noticed that all material shows remarkable resistance to the high stress with longer than 60h for 02% creep for DB3 aged at 900°C for 24h.

Table 6: Creep Data for 02K5 and 02DB3 under a constant stress condition, showing creep life for the very initial creep deformation.

Alloy	History	T (°F)	Stress (MPa)	Creep (%) / Time (h)
02K5B2	900°C/48h/500°C/AC	1400	276	01/15.4; 0.2/54.0
02DB3	900°C/24h/500°C/AC	1400	276	01/11.5; 0.2/64.0
02DB3	900°C/48h/500°C/AC	1400	276	0.1/33.0; 0.2/33.0

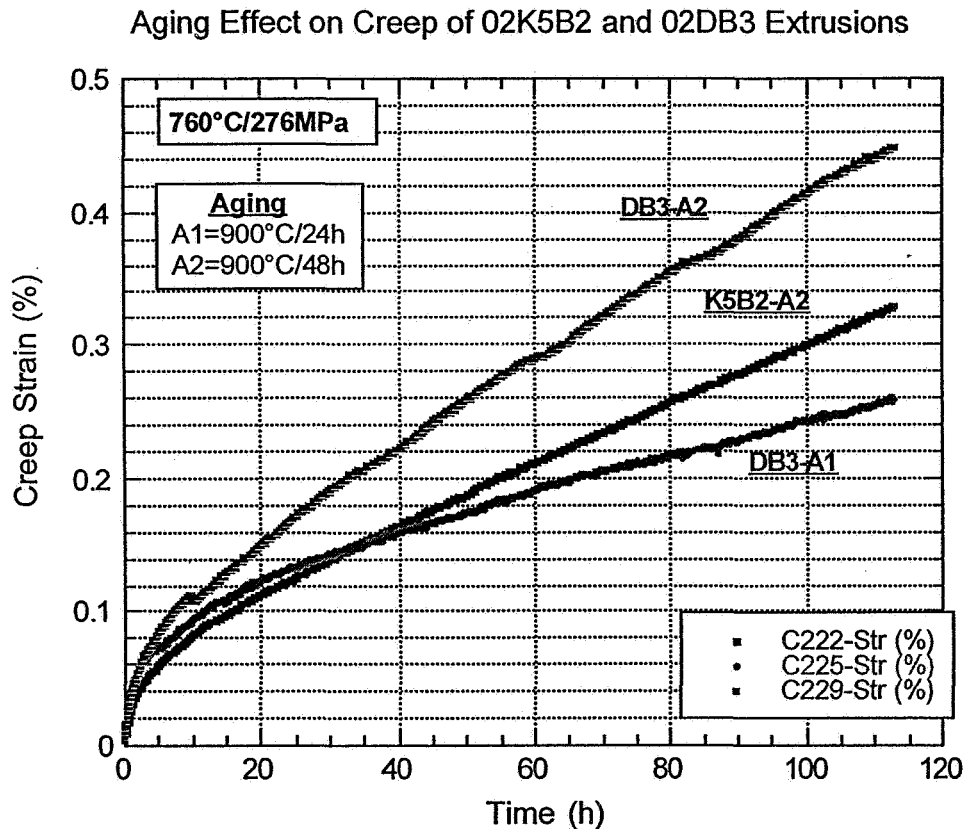


Figure 18: Creep curves for DB3 extrusions aged in two conditions tested under 276MPa and in air, showing primary creep deformation behavior.

Fig. 19 shows the minimum creep rates measured from KB2 and DB3 specimens, aged at 900°C for 48h, as a function of stress applied at 760°C in air. The difference in the variations of  $n$  values between KB2 and DB3 specimens are striking. The min creep rate variations with stress in DB3 specimens is remarkably insensitive compared with that of KB2. This implies that DB3 c

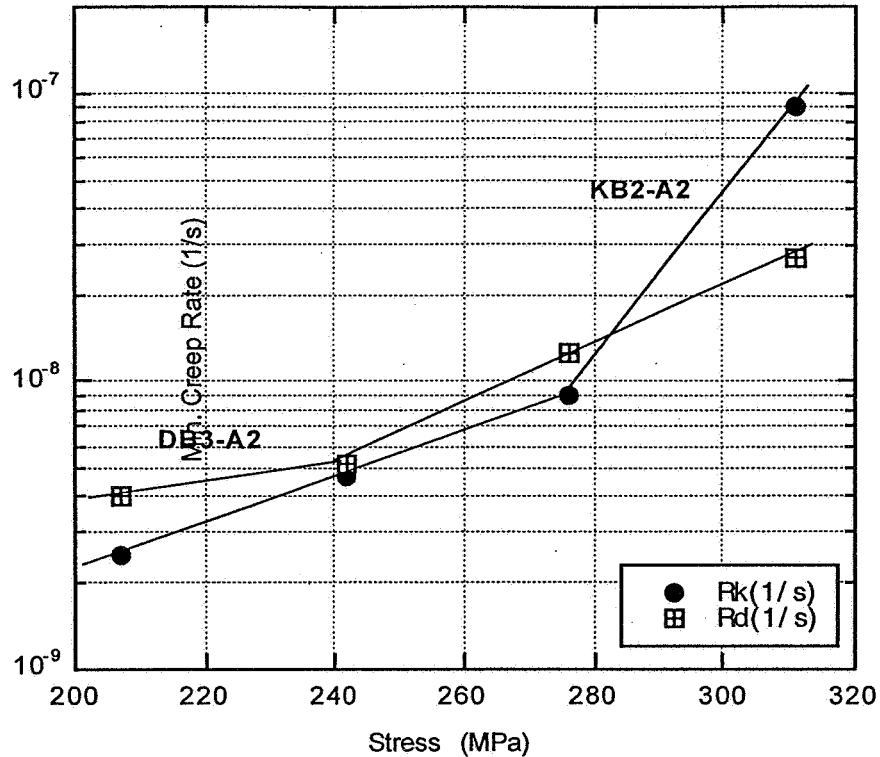



Figure 19: The relations between the minimum creep rate and stress for DB3 and KB2 aged at 900°C for 48h were obtained at 760°C in air. DB3 specimens show a relatively insensitive relationship as compared with KB2 material.

## Summary and Suggestion of Future Work

1. First batch alloy ingots, 02K5B1 and 02DB1, were found contain large inclusions of graphite and Ti-oxides. Nevertheless, their alpha extrusions at extrusion ratio of 18:1, when aged at 900°C, show reasonable ductility levels (2.6% for 02K5B1 and 1.2% for 02DB1) and very high strengths ranging from 600MPa to over 1000MPa for yield strength. 02K5B1 extrusions show lower strength abut and greater ductility. For both alloy extrusions, their microstructures are essentially fully lamellar, with relatively uniform grain sizes below 70  $\mu\text{m}$ .
2. The second batch alloys, 02K5B2 and 02DB3, have not shown to contain inclusions. However, obtaining uniform microstructures throughout the extrusion is difficult, and this needs to be addressed. Their extruded microstructures are similar to their respective counterparts of the first batch alloys. Tensile testing shows that both materials are characterized by high strengths, but KB2 material is less stronger but more ductile as was the case for the first alloys. An exposure (800°C/200h) reduced the RT plasticity nearly



completely for DB3 specimens, whereas KB2 specimens still retain reasonable ductility levels. Fatigue properties are excellent but the non-uniformity of the microstructure is an issue, as was observed in BB alloy specimens. Remarkable creep resistance was observed for both alloys; however, there should be optimum process and aging conditions that will further improve the resistance. It is important to notice that KB2 alloy appears to exhibit the primary creep resistance equal to (at the very early stages) but better than (at later stages) that of alloy DB3 under 276MPa at 760°C.

3. Future work has to be concentrated on producing uniform microstructures, and this will be difficult and serious especially for and in scaled-up shape extrusion, which is anticipated to be the main part of the next program. PM routes can be a solution. So far only a few combinations of annealing and aging conditions have been tested. Testing the materials in more extensive heat treatment conditions is needed to understand the composition-specific processing/aging/structure/property relationships. This effort is time-consuming but essential to design and fix the alloy composition and materials conditions.

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